

# PATENT ABSTRACTS OF JAPAN

(11)Publication number : 2000-178368

(43)Date of publication of application : 27.06.2000

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(51)Int.Cl.

C08J 5/18  
C08G 69/26  
C08L 77/06

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(21)Application number : 10-362060

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(22)Date of filing : 21.12.1998

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## (54) POLYAMIDE SHEET

(57)Abstract:

**PROBLEM TO BE SOLVED:** To provide a novel polyamide sheet which is excellent in all of the performances including heat resistance, chemical resistance, and low water absorption as compared with a conventional polyamide sheet (film).

**SOLUTION:** This sheet is obtained by extruding a polyamide comprising dicarboxylic acid units containing 60-100 mol% terephthalic acid units and diamine units containing 60-100 mol% 9-12C aliphatic alkylenediamine units and having a limiting viscosity number  $[\eta]$  of 1.1-2.5 dl/g as measured in concentrated sulfuric acid at 30° C.

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## LEGAL STATUS

[Date of request for examination] 25.03.2005

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

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EXAMPLE

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[Example] Hereafter, although an example explains this invention concretely, this invention is not restricted at all by these. In addition, the limiting viscosity in an example [eta], the melting point, an elevated-temperature elastic modulus, chemical resistance, and water absorption were measured by the following approaches, respectively.

[0027] 1. Limiting Viscosity [Eta]

Among concentrated sulfuric acid, it asked for the intrinsic viscosity ( $\eta_{inh}$ ) of the sample of each concentration of 0.05, 0.1, 0.2, and 0.4 g/dl from the following formulas at 30 degrees C, and the value which extrapolated this to concentration 0 was made into limiting viscosity [eta].

$\eta_{inh}$  expresses intrinsic viscosity (dl/g) among an  $\eta_{inh} = [\ln(t_1/t_0)]/c$  [type,  $t_0$  expresses the flowing-down time amount (second) of a solvent,  $t_1$  expresses the flowing-down time amount (second) of the sample solution, and  $c$  expresses the concentration (g/dl) of the sample in a solution. ]

[0028] 2. After heating resin for 2 minutes and dissolving it completely at 350 degrees C under a nitrogen air current in the furnace of DSC using a melting point differential scanning calorimeter (product made from METORA "DSC30"), it cooled to 50 degrees C the rate for 100-degree-C/, the location of the endoergic peak which appears when a temperature up is carried out the rate for 10-degree-C/again was measured, and this was made into the melting point.

[0029] 3. The elevated-temperature modulus-of-elasticity dynamic viscoelasticity measuring device ("LEO spectra DVE-V4" by the rheology company) was used, and the storage modulus of the sheet in 250 degrees C was measured.

[0030] 4. The appearance change after a chemical-resistant 4cmx4cm sheet is immersed for seven days into [ various ] a chemical (a 23 degrees C methanol, 23 degrees C 10% sulfuric acid, 23 degrees C 50% sodium-hydroxide water solution, 80-degree C hot water) was observed.

[0031] 5. The saturation water absorption when a water absorption 4cmx4cm sheet being immersed in 23-degree C distilled water was searched for by the gravimetry.

[0032] The example 1 of reference [manufacture of a polyamide]

terephthalic-acid 3272. -- the nitrogen purge of 9g (19. 80 mols), 1, 1582.9 g (10. zero mol) 9-nonane diamine, the 2-methyl -1, 8-octane diamine 1582.9 (10. zero mol), a 48.85 g (0.40 mols) benzoic acid, 6.5g (it is 0.1 % of the weight to the polyamide raw material 4 aforementioned person's sum total) of sodium hypophosphite monohydrates, and the 2.2l. of distilled water was put in and carried out to the autoclave of 20l. of content volume. After stirring for 30 minutes at 100 degrees C, the temperature up of the internal temperature was carried out to 210 degrees C over 2 hours. At this time, the pressure up of the autoclave was carried out to 22kg/cm<sup>2</sup>. A temperature up is carried out to 230 degrees C, and it was made to react after that for 2 hours, after continuing a reaction then for 1 hour, keeping temperature at 230 degrees C, extracting a steam gradually, and keeping a pressure at 22kg/cm<sup>2</sup>. Next, lowered the pressure to 10kg/cm<sup>2</sup> over 30 minutes, it was made to react for further 1 hour, and limiting viscosity [eta] obtained the prepolymer of 0.30 dl/g. After drying this under reduced pressure at the temperature of 100 degrees C for 12 hours, it ground to the magnitude of 2mm or less. Subsequently, solid state polymerization of this was carried out under the temperature of 230 degrees C, and the conditions of pressure 0.1mmHg for 10 hours, and the polyamide of the melting point of 265 degrees C and the white of [limiting viscosity eta] 1.45 dl/g was obtained.

[0033] The examples 2-8 of reference [manufacture of a polyamide]

It used, respectively at a rate which showed the dicarboxylic acid component, the diamine component, and end encapsulant (benzoic acid) which were shown in Table 1 in Table 1, and the polyamide was manufactured by the same approach as the example 1 of reference. The limiting viscosity [eta] and the melting point of a polyamide which were obtained are shown in Table 1.

[0034]

[Table 1]

	ポリアミド原料						ポリアミド物性		ポリアミド 略 称
	ジカルボン酸成分		ジアミン成分		末端封止剤		極限粘度 〔η〕 (dl/g)	融点 (℃)	
	種類	モル数 (mol)	種類	モル数 (mol)	種類	モル数 (mol)			
参考例 1	TA	19.8	NMDA/MODA	10.0/10.0	BA	0.40	1.46	265	9M-T-1
参考例 2	TA	19.8	NMDA/MODA	14.0/8.0	BA	0.40	1.41	285	9M-T-2
参考例 3	NA/TA	3.9/15.9	NMDA	20.0	BA	0.40	1.39	285	9-NT
参考例 4	IA/TA	3.9/15.9	NMDA/MODA	17.0/3.0	BA	0.40	1.43	278	9M-IT
参考例 5	IA/TA	3.9/15.9	DDMDA	20.0	BA	0.20	1.42	290	12-IT
参考例 6	IA/TA	5.9/13.9	EDMDA	20.0	BA	0.40	1.13	320	6-IT
参考例 7	TA	19.8	NMDA/MODA	10.0/10.0	BA	1.40	0.70	265	9M-T-3
参考例 8	TA	20.0	NMDA/MODA	10.0/10.0	-	0	2.60	255	9M-T-4

[略号の内容]

TA : テレフタル酸

IA : イソフタル酸

NA : 2, 6-ナフタレンジカルボン酸

NMDA : 1, 9-ノナンジアミン

MODA : 2-メチル-1, 8-オクタンジアミン

DDMDA : 1, 12-ドデカンジアミン

EDMDA : 1, 6-ヘキサンジアミン

BA : 安息香酸

[0035] The polyamide of examples 1-5 and the examples 1-6 of example of comparison 2 reference was supplied to the biaxial extruder (made in an Oriental energy machine factory "lab PURASUTO mill 2D25W"), and heating fusion was carried out at temperature higher 10-40 degrees C than the melting point of each polyamide, it extruded from the T die, and the polyamide sheet with a width of face [ of 50mm ] and a thickness of 2mm was manufactured. Next, each item shown in Table 2 was evaluated using the obtained polyamide sheet.

[0036] The sheet was manufactured by the approach of examples 1-5 and the examples 1-2 of a comparison, and the same approach, using commercial nylon 6 ("Amilan CM1021XF" by Toray Industries) as example of comparison 1 polyamide. Thus, each item shown in Table 2 was evaluated using the obtained sheet.

[0037] Although the polyamide of the example 7 of example of comparison 3 reference was supplied to the biaxial extruder (made in an Oriental energy machine factory "lab PURASUTO mill 2D25W"), and heating fusion was carried out at 280 degrees C - 290 degrees C and extruded from the T die, viscosity was low and a sheet was not able to be manufactured.

[0038] Although the polyamide of the example 8 of example of comparison 4 reference was supplied to the biaxial extruder (made in an Oriental energy machine factory "lab PURASUTO mill 2D25W"), and heating fusion was carried out at 280 degrees C - 290 degrees C and extruded from the T die, viscosity was high and a sheet was not able to be manufactured.

[0039]

[Table 2]

	実施例 1	実施例 2	実施例 3	実施例 4	実施例 5	比較例 1	比較例 2	比較例 3	比較例 4
ポリアミド	9M-T-1	9M-T-2	9-NT	9M-IT	12-IT	ナイロン 6	6-IT	9M-T-3	9M-T-4
	参考例 1	参考例 2	参考例 3	参考例 4	参考例 5	—	参考例 6	参考例 7	参考例 8
高弾性率 E' (250℃) 10 <sup>10</sup> dyn/cm <sup>2</sup>	1. 2	1. 7	1. 4	1. 6	1. 0	検 動	0. 3	* 1	* 1
耐薬品性 30% 塩酸 10% 硝酸 50% NaOH 80℃ 熱水	変化なし 変化なし 変化なし 変化なし	変化なし 変化なし 変化なし 変化なし	変化なし 変化なし 変化なし 変化なし	変化なし 変化なし 変化なし 変化なし	変化なし 変化なし 変化なし 変化なし	変形大 一部溶解 変化なし 白化	白化 白化 変化なし 白化	* 1	* 1
吸水率 (28℃水中飽和)	3. 3	2. 9	2. 9	3. 1	2. 5	12. 1	6. 9	* 1	* 1

\* 1 : シートが得られなかった。

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**MEANS**

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[Means for Solving the Problem] In order to solve the above-mentioned technical problem, as a result of inquiring wholeheartedly, this invention persons are polyamides which use aliphatic series alkylene diamine of a terephthalic acid and carbon numbers 9-12 as a principal component, find out that the sheet with which it will not be satisfied without by producing an extrusion-molding sheet of the above-mentioned purpose is obtained from the polyamide which has specific limiting viscosity [eta], and came to complete this invention.

[0008] namely, this invention -- a terephthalic-acid unit -- 60-100-mol % -- the dicarboxylic acid unit to contain and the aliphatic series alkylene diamine unit of carbon numbers 9-12 -- 60-100-mol % -- it consists of a diamine unit to contain and is related with the sheet with which the limiting viscosity [eta] measured at 30 degrees C among concentrated sulfuric acid carries out extrusion molding of the polyamide which is 1.1 - 2.5 dl/g, and is obtained.

[0009]

[Embodiment of the Invention] Hereafter, this invention is explained to a detail. as the dicarboxylic acid unit which constitutes the polyamide used for this invention -- a terephthalic-acid unit -- 60-100-mol % -- it contains -- \*\*\*\* -- 70-100-mol % -- containing is desirable. When a terephthalic-acid unit is less than [ 60 mol % ], the thermal resistance of the polyamide sheet obtained and low absorptivity fall.

[0010] As other dicarboxylic acid units other than a terephthalic-acid unit A malonic acid, a succinic acid, a glutaric acid, an adipic acid, a pimelic acid, a suberic acid, An azelaic acid, a sebacic acid, dodecane dicarboxylic acid, a dimethyl malonic acid, 3 and 3-diethyl succinic-acid, 2, and 2-dimethyl glutaric acid, 2-methyl adipic acid, Aliphatic series dicarboxylic acid, such as a trimethyl adipic acid; 1, 3-cyclopentane dicarboxylic acid, Alicyclic dicarboxylic acid, such as 1 and 4-cyclohexane dicarboxylic acid; Isophthalic acid, 2, 6-naphthalene dicarboxylic acid, 2, 7-naphthalene dicarboxylic acid, 1, 4-naphthalene dicarboxylic acid, 1, 4-phenylene dioxy diacetate, 1,3-phenylenedioxy diacetate, diphenic acid, 4, and 4'-biphenyl dicarboxylic acid, The unit guided from aromatic series dicarboxylic acid, such as 4, a 4'-oxydi benzoic acid, diphenylmethane -4, 4'-dicarboxylic acid, diphenylsulfone -4, and 4'-dicarboxylic acid, can be mentioned. These units can use one sort or two sorts or more. Also in these, the unit guided from alicyclic dicarboxylic acid, such as aromatic series dicarboxylic acid [ , such as 2, 6-naphthalene dicarboxylic acid, and isophthalic acid ], 1, and 4-cyclohexane dicarboxylic acid, is desirable. Furthermore, the unit guided from polyvalent carboxylic acid, such as trimellitic acid, trimesic acid, and pyromellitic acid, can also be made to contain within limits with melting shaping of a polyamide possible in which.

[0011] as the diamine unit which constitutes the polyamide used for this invention -- the aliphatic series alkylene diamine unit of carbon numbers 9-12 -- 60-100-mol % -- it contains -- \*\*\*\* -- 75-100-mol % -- containing -- desirable -- 90-100-mol % -- containing is more desirable. When the content of the aliphatic series alkylene diamine unit of carbon numbers 9-12 is less than [ 60 mol % ], the thermal resistance of the polyamide sheet obtained, low absorptivity, and chemical resistance fall. As an aliphatic series alkylene diamine unit of these carbon numbers 9-12 For example, 1, 9-nonane diamine, 1, 10-Decan diamine, 1, and 11-undecane diamine, Straight chain-like aliphatic series alkylene diamines, such as 1 and 12-dodecane diamine; 2, 2, 4-trimethyl -1, 6-hexanediamine, 2, 4, and 4-trimethyl -1, 6-hexanediamine, 2, the 4-diethyl -1, 6-hexanediamine, The 2 and 2-dimethyl -1, 7-heptane diamine, 2, the 3-dimethyl -1, 7-heptane diamine, 2, the 4-dimethyl -1, 7-heptane diamine, 2, the 5-dimethyl -1, 7-heptane diamine, The 2-methyl -1, 8-octane diamine, the 3-methyl -1, 8-octane diamine, The 4-methyl -1, 8-octane diamine, 1, the 3-dimethyl -1, 8-octane diamine, 1, the 4-dimethyl -1, 8-octane diamine, 2, the 4-dimethyl -1, 8-octane diamine, 3, the 4-dimethyl -1, 8-octane diamine, 4, the 5-dimethyl -1, 8-octane diamine, The 2 and 2-dimethyl -1, 8-octane diamine, 3, and 3-dimethyl -1, 8-octane diamine, Branched-chain aliphatic series alkylene diamines, such as the 4 and 4-dimethyl -1, 8-octane diamine, the 5-methyl -1, and 9-nonane diamine; Norbornane dimethylamine, The unit guided from alicyclic diamines, such as isophorone diamine and tricyclodecane dimethylamine, etc. can be mentioned, and one sort or two sorts or more can be used among these.

[0012] In the above-mentioned aliphatic series alkylene diamine unit, the unit guided from 2-methyl -1, 8-octane diamine, 1, 9-nonane diamine, 1, 10-Decan diamine, 1, and 11-undecane diamine and 1, and 12-dodecane diamine is desirable, and 1 and 9-nonane diamine (NMDA) unit and the 2-methyl -1, and 8-octane diamine (MODA) unit are more desirable.

Suitably, it is desirable that a NMDA unit:MODA unit uses a NMDA unit and a MODA unit also in 100:0-30:70 (mole ratio) at 99:1-40:60, and a rate that is set especially to 98:2-45:55. By using a NMDA unit and a MODA unit together at an above-mentioned rate, the sheet excellent in thermal resistance, a moldability, and low absorptivity is obtained especially from such a polyamide.

[0013] As a diamine unit which can be used in addition to the aliphatic series alkylene diamine unit of the above-mentioned carbon numbers 9-12 For example, aliphatic series diamines, such as ethylenediamine, propylenediamine, and 1,4-butanediamine; A cyclohexanediamine, Alicyclic diamines, such as methylcyclohexane diamine; P-phenylene diamine, m-phenylenediamine, p-xylylene diamine, m-xylylene diamine, The unit guided from aromatic series diamines, such as - diaminodiphenyl sulfone, and 4 and 4'-diamino diphenylmethane, 4, and 4 '4, 4'-diamino diphenyl ether, etc. can be mentioned, and one sort or two sorts or more can be used among these.

[0014] The end group of the chain is desirable and, as for the polyamide used for this invention, the closure of the 70% or more is especially carried out with end encapsulant preferably 40% or more 10% or more. If the rate of the end closure is 10% or more, the viscosity change at the time of melting shaping of a polyamide is small, and since physical properties, such as an appearance of the sheet obtained and hot water resistance, are excellent, it is desirable. In asking for the rate of the closure of an end, the number of the ends by which the closure was carried out with the carboxyl group end, the amino-group end, and end encapsulant which exist in the polyamide can be measured, respectively, and it can ask for the rate of the closure of an end by the following formula (1). As for the number of each end groups, it is desirable to ask from the integral value of the property signal corresponding to each end group by <sup>1</sup> H-NMR in respect of precision and simplicity.

[0015]

Rate (%) of the end closure =  $[(A-B) / A] \times 100$  (1)

A expresses a chain end group total (this is usually equal the twice of the number of polyamide molecules) among [type, and B expresses the total number of a carboxyl group end and an amino-group end. ]

[0016] Although there is especially no limit and acid anhydrides, such as monocarboxylic acid, monoamine, and phthalic anhydride, mono-isocyanate, mono-acid halide, monoester, and monoalcohol can be used if it is the compound of the monofunctional nature which has the amino group of a polyamide end or a carboxyl group, and reactivity as end encapsulant, a point to monocarboxylic acid or monoamines, such as reactivity and the stability of a closure end, are desirable, and monocarboxylic acid is more desirable from points, such as an ease of handling.

[0017] As monocarboxylic acid used as end encapsulant If it has reactivity with the amino group, although there will be especially no limit For example, an acetic acid, a propionic acid, butanoic acid, a valeric acid, a caproic acid, a caprylic acid, A lauric acid, a tridecyl acid, a myristic acid, a palmitic acid, stearic acid, Alicyclic monocarboxylic acid [ , such as aliphatic series monocarboxylic acid; cyclohexane carboxylic acid ], such as pivalate and an isobutyl acid; A benzoic acid, Aromatic series monocarboxylic acid, such as a toluic acid, alpha-naphthalene carboxylic acid, beta-naphthalene carboxylic acid, a methylnaphthalene carboxylic acid, and a phenylacetic acid, etc. can be mentioned. These can use one sort or two sorts or more. Also in these, the acetic acid from points, such as reactivity, the stability of a closure end, and a price, a propionic acid, butanoic acid, a valeric acid, a caproic acid, a caprylic acid, a lauric acid, a tridecyl acid, a myristic acid, a palmitic acid, stearic acid, and a benzoic acid are desirable.

[0018] Although there will be especially no limit as monoamine used as end encapsulant if it has reactivity with a carboxyl group, aromatic series monoamines, such as alicyclic monoamine; anilines, such as aliphatic series monoamine; cyclohexylamines, such as monomethylamine, ethylamine, propylamine, a butylamine, hexylamine, an octyl amine, a DESHIRU amine, a stearyl amine, dimethylamine, diethylamine, a dipropyl amine, and dibutyl amine, and dicyclohexylamine, a toluidine, a diphenylamine, and a naphthylamine, etc. can be mentioned, for example. These can use one sort or two sorts or more. Also in these, the butylamine from points, such as the stability of reactivity, the boiling point, and a closure end and a price, hexylamine, an octyl amine, a DESHIRU amine, a stearyl amine, cyclohexylamine, and an aniline are desirable.

[0019] The polyamide used for this invention has more preferably the limiting viscosity [eta] measured at 30 degrees C among concentrated sulfuric acid within the limits of 1.2 - 1.8 dl/g within the limits of 1.15 - 2.0 dl/g within the limits of 1.1 - 2.5 dl/g. If [ above-mentioned ] the limiting viscosity [eta] of a polyamide is within the limits, while it is excellent in the moldability to a sheet, the sheet excellent in kinetic property, thermal resistance, etc. is obtained.

[0020] As for the polyamide used for this invention, it is desirable that a consistency is less than 1.16. If a consistency is this range, since the sheet excellent in low absorptivity, chemical resistance, and toughness is obtained, it is desirable.

[0021] Moreover, in order to raise more many properties of the sheet finally obtained to the polyamide used for this invention Within limits for which the moldability to the sheet of this invention or many properties as a sheet are not sacrificed The need is accepted. A coloring agent; ultraviolet ray absorbent; light stabilizing agent; hindered phenol system, Organic system antioxidants, such as a thio system, the Lynn system, and an amine system; Copper iodide, copper acetate, inorganic system antioxidant [ , such as potassium iodide, ]; -- antistatic-agent; -- fluorescent brightener; -- a bromination polymer -- Flame-retarder; plasticizers, such as antimony oxide and a metal hydroxide; lubricant ;P Other

type polymers, such as PS, a liquid crystal polymer, polyolefine, polyester, an aliphatic series polyamide, and PPO; the various powdered or fibrous bulking agents of an organic system and an inorganic system can also be added.

[0022] As the addition approach of the various above-mentioned additives, dryblend is carried out to the approach and polyamide which are added at the time of the polymerization of a polyamide, for example, and the approach of carrying out melting kneading, the approach of adding at the time of extrusion molding to a sheet, etc. are adopted.

[0023] The polyamide sheet of this invention is manufactured by \*\*\*\*\* which gives the above-mentioned polyamide to the T-die extrusion method to which it is usually applied by manufacture of a sheet. For example, after carrying out melting of the above-mentioned polyamide at the temperature of 370 degrees C or less more than the melting point of this polyamide with an extruder, it is fabricated on a sheet thicker than 200 micrometers by extruding by the T die and usually cooling on a sticking-by-pressure roll.

[0024] A front face can be used for the polyamide sheet of this invention also where a laminating is carried out to a coating, a metal layer, an other type polymer, etc.

[0025] the description which the polyamide sheet of this invention does not have in the conventional polyamide sheet (film) -- have -- \*\*\*\* -- dielectric-materials [, such as electrical insulation material; capacitor applications, such as a motor, a transformer, and a cable, ]; -- charge; medical-application ingredient; engineering works [, such as a magnetic material; signboard, ] of ornament ingredient; agricultural material, such as electronic substrate ingredient; sheet-like magnets, such as a printed circuit board and a flat cable, a structural ingredient; filtration membrane, etc. can be suitably use as a sheet for home use and industrial materials.

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TECHNICAL PROBLEM

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[Problem(s) to be Solved by the Invention] According to research of this invention persons, when using it as an industrial ingredient since a dimensional change and a fall on the strength are large since water absorption is high, and the endurance over alcohol, an acid, alkali, etc. is not enough although thermal resistance of the polyamide film indicated by JP,2-248433,A and JP,3-253324,A is improving as compared with the conventional polyamide film, an application is restricted considerably.

[0006] The purpose of this invention is to offer the polyamide sheet which was excellent in thermal resistance, chemical resistance, and any engine performance of low absorptivity as compared with the conventional polyamide film (sheet).

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EFFECT OF THE INVENTION

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[Effect of the Invention] The polyamide sheet of this invention is excellent in thermal resistance, chemical resistance, and any engine performance of low absorptivity as compared with the conventional polyamide sheet (film), and can be used suitable for the application of industrial materials, an industrial ingredient, household articles, etc.

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PRIOR ART

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[Description of the Prior Art] Since it has the outstanding toughness, thermal resistance, cold resistance, a printing property, chemical resistance, etc., the polyamide sheet (film) is used as wrapping or an industrial ingredient. However, the demand of a commercial scene is developed, and it is thermal resistance more, and excels in the bottom of an elevated temperature, or the dimensional stability under highly humid, and the demand on the sheet with high endurance to a chemical is increasing every year.

[0003] The polyamide film which makes the main constituents half-aromatic polyamide (it may be called PA6<sup>6T</sup> for short below) which consists of all the aliphatic series polyamides (it may call for short the following PA 4-6) and terephthalic acid which consist of an adipic acid and 1,4-butanediamine, and a 1 and 6-hexanediamine is proposed to the demand of such a world.

[0004] For example, it is indicated by JP,2-248433,A that the polyamide film with which the rate of PA4-6:half aromatic polyamide consists of a polyamide constituent which is 99.9:0.1-30:70 is excellent in thermal resistance. Moreover, it is indicated by JP,3-253324,A that the polyamide film which uses PA6-T as a principal component is excellent in gas barrier nature, reinforcement, thermal resistance, and transparency.

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TECHNICAL FIELD

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[Field of the Invention] This invention relates to a half-aromatic polyamide sheet. In detail, it is related with the half-aromatic polyamide sheet excellent in thermal resistance, chemical resistance, and any engine performance of low absorptivity.

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CLAIMS

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[Claim(s)]

[Claim 1] a terephthalic-acid unit -- 60-100-mol % -- the dicarboxylic acid unit to contain and the aliphatic series alkylene diamine unit of carbon numbers 9-12 -- 60-100-mol % -- the sheet with which the limiting viscosity  $[\eta]$  which consisted of a diamine unit to contain and was measured at 30 degrees C among concentrated sulfuric acid carries out extrusion molding of the polyamide which is 1.1 - 2.5 dl/g, and is obtained.

[Claim 2] The sheet according to claim 1 whose mole ratios of 1, the 9-nonane diamine unit:2-methyl -1, and 8-octane diamine unit the aliphatic series alkylene diamine unit of carbon numbers 9-12 consists of 1 and 9-nonane diamine unit and 2-methyl -1, and a 8-octane diamine unit, and are 100:0-30:70.

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